IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

.....

Serial No: 10/575,599
Filed: April 13, 2006
Title: DISPERSE AZO DYESTUFFS

Art Unit: 1626
Examiner: Fiona Powers

.....

Hon. Commissioner of Patents & Trademarks
Washington, D. C. 20 231

DECLARATION (Rule 132)

Sir:

I, Adrian Murgatroyd from Rossendale, UK, declare: I am a Chemical Engineer and a citizen of the United Kingdom, residing at Wed 9, 65929 Frankfurt am Main, Federal Republic of Germany.

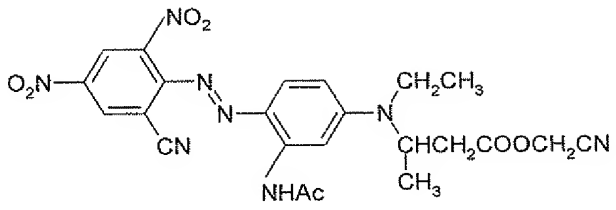
Since completing my studies at the University of Exeter in the United Kingdom, I have been employed as a textile technician by Tootal Limited, Manchester, UK and as a development manager by ICI (subsequently Zeneca), Manchester, UK. The textile activities of Zeneca were taken over by BASF Aktiengesellschaft, Ludwigshafen, Germany, where I worked as a product manager and as a development manager. In October 2000 BASF transferred its activities in the textile dyestuff field to DyStar and since then I have been employed by DyStar Textilfarben GmbH & Co. Deutschland KG in Frankfurt, Germany as a product development manager for disperse dyes.

I have had adequate professional experience in the field to which patent application Serial No. 10/575,599, filed April 13, 2006, pertains and which was filed by Nigel Hall.

I further declare:
In order to demonstrate that the dyestuffs according to the present application are not obvious over the teachings of the prior art the tests described below have been carried out under my personal guidance and supervision.

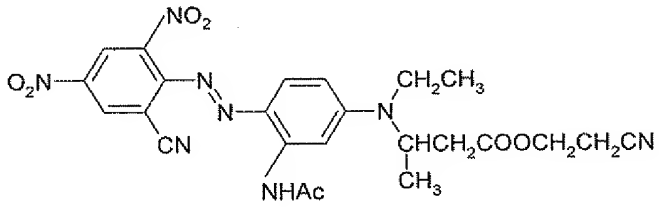
I. DYESTUFFS

1. Dyestuff 5 of the formula



according to the present invention

2. Dyestuff 6 of the formula



according to Example 20 of GB 2,104,088 to Dawson et al. (prior art)

II) DETERMINATION OF THE DYESTUFFS' COLOUR FASTNESS (AATCC Test Method 61-2003)

Dyestuffs 5 and 6 were dyed on a 100% polyester woven fabric at concentrations adjusted to give equal visual depths (Integ 30 or 1.5 times standard depth). The dyeings were subsequently reduction cleared, dried and then post-set at 180°C for 30 seconds.
The dyeings obtained with Dyestuffs 5 and 6, respectively, were then subjected to the AATCC Test Method 61-2003 "Colorfastness to Laundering, Home and Commercial: Accelerated" Test Number 2A (see copy enclosed). The results are given on the enclosed Shade Card.

III. RESULTS

The tests show that the dyeings obtained with the inventive Dyestuff 5 show a significantly

higher fastness to the AATCC test when compared to the dyeings obtained with Dyestuff 6 according to prior art. Dyeings obtained with Dyestuff 5 show staining on both polyester and polyamide adjacents (multifibre strip) of 4 rated by comparison with the grey scale, whereas dyeing obtained with Dyestuff 6 show staining on both polyester and polyamide adjacents at a grey scale level of 3. This improvement could not at all be foreseen for a person skilled in the art and was thus unexpected and surprising.

I further declare that I understand the contents of this Declaration, that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Frankfurt
This 20th day of November 2007

Adrian Murgatroyd

(Adrian Murgatroyd)

Colorfastness to Laundering, Home and Commercial: Accelerated

Developed in 1950 by AATCC Committee RA60; revised 1952, 1954, 1957, 1960, 1961, 1970, 1972, 1986 (title change), 1989, 1993, 1994, 1996, 2003; reaffirmed 1956, 1962, 1965, 1968, 1969, 1975, 1980, 1985; editorially revised 1973, 1974, 1975, 1976, 1981, 1983, 1984, 1991, 1995, 1998, 2002, 2004; editorially revised and reaffirmed 2001. Partly equivalent to ISO 105-C06.

1. Purpose and Scope

1.1 These accelerated laundering tests are to evaluate the colorfastness to laundering of textiles which are expected to withstand frequent laundering. The fabric color loss and surface changes resulting from detergent solution and abrasive action of five typical hand, home or commercial launderings, with or without chlorine, are roughly approximated by one 45 min test (see 9.2-9.6). However, the staining effect produced by five typical hand, home or commercial launderings cannot always be predicted by the 45 min test. Staining is a function of the ratio of colored to undyed fabrics, fiber content of fabrics in the wash load and other end-use conditions which are not always predictable.

2. Principle

2.1 Specimens are tested under appropriate conditions of temperature, detergent solution, bleaching and abrasive action such that the color change is similar to that occurring in five hand, home or commercial launderings. The color change is obtained in a conveniently short time. The abrasive action is a result of the frictional effects of fabric against canister, the low liquor ratio and the impact of the steel balls on the fabric.

3. Terminology

3.1 **colorfastness,** n.—the resistance of a material to change in any of its color characteristics, to transfer of its colorant(s) to adjacent materials or both, as a result of the exposure of the material to any environment that might be encountered during the processing, testing, storage or use of the material.

3.2 **laundering,** n.—*of textile materials*, a process intended to remove soils and/or stains by treatment (washing) with an aqueous detergent solution and normally including subsequent rinsing, extracting and drying.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in the test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 The 1993 AATCC Standard Reference Detergent WOB may cause irritation. Care should be taken to prevent exposure to skin and eyes.

4.4 An eyewash/safety shower should be located nearby for emergency use.

4.5 Manufacturer's safety recommendations should be followed when operating laboratory testing equipment.

5. Apparatus, Reagents and Materials

5.1 Launder-Ometer.

5.1.1 A laundering machine for rotating closed canisters in a thermostatically controlled water bath at 40 ± 2 rpm (see 12.1).

5.1.2 Stainless steel lever lock canisters Type 1 (see 12.1) 500 mL (1 pt), 75 × 125 mm (3.0 × 5.0 in.) for Test No. 1A.

5.1.3 Stainless steel lever lock canisters Type 2 (see 12.1) 1200 mL, 90 × 200 mm (3.5 × 8.0 in.) for Tests No. 2A, 3A, 4A and 5A.

5.1.4 Adapter plates for holding canisters (see 5.1.3) on laundering machine shaft (see 12.1).

5.1.5 Stainless steel balls, 6 mm (0.25 in.) in diameter (see 12.1).

5.1.6 Teflon fluorocarbon gaskets (see 7.4.2, 12.1 and 12.2).

5.1.7 Preheater/storage module (see 7.4, 12.1 and 12.3).

5.2 Scales for rating test results.

5.2.1 AATCC Chromatic Transference Scale (see 12.4).

5.2.2 Gray Scale for Color Change (see 12.4).

5.2.3 Gray Scale for Staining (see 12.4).

5.3 Reagents and materials.

5.3.1 Multifiber test fabrics No. 1 and FB (8 mm [0.33 in.] bands) contains bands of acetate, cotton, nylon, silk, viscose rayon and wool. Multifiber test fa
rics No. 10, and FA (8 mm [0.33 i
bands) and No. 10A and FAA (15 m
[0.6 in.] bands) contain bands of aceta
cotton, nylon, polyester, acrylic and wc
(see 12.5).

5.3.2 Bleached cotton test fabric, 32
32 ends × picks/cm (80 × 80 ends
picks/in.) construction, 100 ± 3 g/m², d
sized without fluorescent whitening age
(see 12.5).

5.3.3 1993 AATCC Standard Refe
ence Detergent WOB (without fluore
cent whitening agent and without phc
phate) (see 10.5 and 12.6).

5.3.4 1993 AATCC Standard Refe
ence Detergent (with fluorescent white
ing agent) (see 10.5 and 12.6).

5.3.5 Water, distilled or deionized (s
12.7).

5.3.6 Sodium hypochlorite (NaOC
bleach (see 12.8).

5.3.7 Sodium carbonate (Na_2CO_3).

5.3.8 Sulfuric acid (H_2SO_4), 10% (s
12.8.1).

5.3.9 Potassium iodide (KI), 10% (s
12.8.1).

5.3.10 Sodium thiosulfate ($Na_2S_2O_3$
0.1*N* (see 12.8.1).

5.3.11 Crockmeter test cloth cut in
mm (2 in.) squares (see 12.9).

5.3.12 White cards (specimen mount
with *Y* tristimulus value at least 85%.

6. Test Specimens

6.1 The sizes of the specimens require
for the various tests are as follows:

50 × 100 mm (2.0 × 4.0 in.) for Te
No. 1A,

50 × 150 mm (2.0 × 6.0 in.) for Tes
No. 2A, 3A, 4A and 5A.

6.2 Test only one specimen in eac
canister.

6.2.1 Test one specimen per laborato
sample. Replication may be advisable fi
improved precision.

6.3 To determine staining in Tests N
1A and 2A, use multifiber test fabric. T
determine staining in Test No. 3A, use e
ther multifiber test fabric or bleached co
ton test fabric. With respect to Test No. 3
the use of multifiber test fabric is option
but the staining of acetate, nylon, polyest
and acrylic is disregarded unless one
these fibers is present in the fabric bein
tested or known to be in the final garmen
For Test 3A, multifiber test fabric wit
heat-sealed edges is recommended. Stai
ing is not determined in Tests No. 4A an
5A (see 12.10 and 12.11).

6.4 Specimen preparation.

6.4.1 Preparation with multifiber te

fabric with individual component bands 8 mm (0.33 in.) wide or with bleached cotton test fabric. Prepare pieces with a 50 mm (2.0 in.) square of multifiber test fabric cloth or bleached cotton test fabric (as required) sewn, stapled or suitably attached along one 50 mm (2.0 in.) edge of the test specimen and in contact with the face of the material. When multifiber test fabric is used, attach it so that each of the six fiber bands is along the 50 mm (2.0 in.) edge of the specimen with the wool on the right. The fiber bands in the multifiber test fabric will be parallel to the lengthwise direction of the test specimen.

6.4.2 Preparation with multifiber test fabric with individual component bands 15 mm (0.6 in.) wide. Prepare pieces with a 50 × 100 mm (2.0 × 4.0 in.) rectangle of multifiber test fabric sewn, stapled or suitably attached centered along one 100 mm (4.0 in.) or 150 mm (6.0 in.) edge of the test specimen and in contact with the face of the material. Attach it so that each of the six fiber bands will be parallel to the widthwise direction of the specimen. Attach and secure the wool band at the top of the specimen to avoid fiber loss.

6.4.3 It is recommended that knitted fabrics be sewn or stapled at the four edges to equivalent size pieces of bleached cotton test fabric to avoid rolled edges and to assist in obtaining a uniform test result over the entire surface. Attach the multifiber test fabric to the face of the knitted fabric.

6.4.4 For pile fabric specimens with a pile lay direction, attach the multifiber test fabric at the top of the specimen with the pile lay direction pointing away from the top of the specimen.

6.5 When the textile to be tested is yarn, specimens may be tested using Option 1 or Option 2.

6.5.1 Option 1. Knit yarn on an appropriate sample knitting machine. Prepare specimens and multifiber test fabrics according to 6.1-6.4.3. Keep one knitted specimen of each sample as an unwashed original.

6.5.2 Option 2. Prepare two 110 m (120 yd) skeins of each yarn. Fold the skein so that there is a uniform amount of yarn across a 50 mm (2 in.) width with a length appropriate for the procedure to be used. Keep one skein of each sample as an unwashed original. Sew or staple Crockmeter test cloth squares (see 12.9) or squares of bleached cotton test fabric having approximately the same weight folded over each end of the layered yarn specimen. Attach a multifiber test fabric according to 6.4.1 or 6.4.2.

7. Procedure

7.1 Table I summarizes the conditions of the tests.

7.2 Adjust the laundering machine to maintain the designated bath temperature. Prepare the required volume of wash liquor. Preheat this solution to the prescribed temperature.

Table I—Test Conditions[a]

Test No.	Temp °C (±°2)	Temp °F(±°4)	Total Liquor Volume	Percent Detergent of Total Volume	Available Chlorine of Total Volume	No. Steel Balls	Time
1A	40	105	200 mL	0.37%	None	10	45 Min
2A	49	120	150	0.15	None	50	45
3A	71	160	50	0.15	None	100	45
4A	71	160	50	0.15	0.015%	100	45
5A	49	120	150	0.15	0.027	50	45

[a]Refer to Section 9 for objectives for each test method.

7.3 Run Test No. 1A in 75 × 125 mm (3.0 × 5.0 in.) lever lock stainless steel canisters. Run Tests No. 2A, 3A, 4A and 5A in 90 × 200 mm (3.5 × 8.0 in.) lever lock stainless steel canisters.

7.3.1 For Tests No. 1A, 2A and 3A, add to the canister the amount of detergent solution designated in Table I.

7.3.2 For Test No. 4A, prepare a 1500 ppm available chlorine solution. For 1 L, determine the amount of stock sodium hypochlorite bleach solution (see 12.8) to dilute as follows:

$$159.4/\%\ \text{NaOCl} = \text{g to add}$$

Weigh the correct amount of bleach into a volumetric flask and dilute to 1 L. To each canister, add 5 mL of 1500 ppm available chlorine solution and 45 mL of detergent solution making a total volume of 50 mL.

7.3.3 For Test No. 5A, determine the amount of stock sodium hypochlorite bleach solution (see 12.8) to dilute as follows:

$$4.54/\%\ \text{NaOCl} = \text{g to add}$$

Weigh the correct amount of bleach into a graduated cylinder and add detergent solution to make a total volume of 150 mL. Prepare this solution separately for each canister.

7.3.4 For all the tests, add the designated number of stainless steel balls to each canister.

7.4 The two options for preheating the canisters to the test temperature are by use of the laundering machine or the preheater/storage unit. If the canisters are to be preheated in the laundering machine, proceed to 7.4.2.

7.4.1 Place the canisters in the preheater module at the prescribed test temperature. They are to remain in the module for at least 2 min. Enter a well crumpled test specimen into each canister.

7.4.2 Clamp the covers on the canisters. A Teflon fluorocarbon gasket (see 5.1.6) may be inserted between the neoprene gasket and the top of each canister to prevent contamination of the wash solution by the neoprene. Fasten the 75 × 125 mm (3.0 × 5.0 in.) lever lock canisters vertically and the 90 × 200 mm (3.5 × 8.0 in.) lever lock canisters horizontally in the adapters on the rotor of the laundering machine in such a manner that when the canisters rotate, the covers strike the water first. Place an equal number of canisters on each side of the shaft. For canisters preheated in the module, proceed to 7.7.

7.5 Start the rotor and run it for at least 2 min to preheat the canisters.

7.6 Stop the rotor and with a row of canisters in an upright position, unclamp the cover of one canister, enter a well crumpled test specimen into the solution and replace the cover, but do not clamp it. Repeat this operation until all the canisters in the row have been loaded. Then clamp the covers in the same order in which the canisters were loaded (delay clamping the covers to allow equalization of pressure). Repeat this operation until all rows of canisters have been loaded.

7.7 Start the laundering machine and run it at 40 ± 2 rpm for 45 min.

7.8 The rinsing, extracting and drying procedures are the same for all the tests. Stop the machine, remove the canisters and empty the contents into beakers, keeping each test specimen in a separate beaker. Rinse each test specimen three times, in beakers, in distilled or deionized water at 40 ± 3°C (105 ± 5°F) for 1 min periods with occasional stirring or hand squeezing. To remove excess water, centrifuge, blot or pass the test specimens through wringer rolls. Dry the specimens in an air circulating oven in which the temperature does not exceed 71°C (160°F), or tumble dry in a nylon mesh bag in an automatic tumble dryer at normal cycle, which has an exhaust temperature of 60-71°C (140-160°F), or air dry.

7.9 Allow specimens to condition at 65 ± 2% relative humidity and 21 ± 1°C (70 ±2°F) for 1 h before evaluating.

7.10 Prepare tested fabric specimens and adjacent fabrics for evaluation by trimming off raveled yarns and lightly brushing off any loose fiber and yarn on the fabric surfaces. Brush pile fabric specimens in required direction to restore them as nearly as possible to the same pile angle as the untreated specimens. Specimens should be smoothed or flat-

tened if they are wrinkled and messy due to washing and/or drying. Specimens may be mounted on cards to facilitate identification and handling in their evaluation. For consistency in backing material, use a white mounting card with *Y* tristimulus value of at least 85%. Mounting material must not be visible in the area to be viewed and must not interfere with rating as specified in 5.1 of both Evaluation Procedures 1 and 2 for the Gray Scale for Color Change and Gray Scale for Staining or instrumental assessment of color according to AATCC Evaluation Procedure 7, Instrumental Assessment of the Change in Color of a Test Specimen.

7.10.1 Yarn skein specimens should be combed and brushed for improved alignment of the yarns before comparison with the unwashed original. The original specimens may also need further combing and brushing for uniformity of appearance.

8. Evaluation

8.1 Evaluation of color change.

8.1.1 Evaluate the color change of the test specimens as directed in Evaluation Procedure 1 using the Gray Scale for Color Change. For improved precision and accuracy the specimens should be rated by more than one rater.

Grade 5—negligible or no change as shown in Gray Scale Step 5.

Grade 4.5—change in color equivalent to Gray Scale Step 4.5.

Grade 4—change in color equivalent to Gray Scale Step 4.

Grade 3.5—a change in color equivalent to Gray Scale Step 3.5.

Grade 3—change in color equivalent to Gray Scale Step 3.

Grade 2.5—change in color equivalent to Gray Scale Step 2.5.

Grade 2—change in color equivalent to Gray Scale Step 2.

Grade 1.5—change in color equivalent to Gray Scale Step 1.5.

Grade 1—change in color equivalent to Gray Scale Step 1.

8.1.2 The color change can be quantitatively determined by measuring the color difference between the unwashed sample and a test specimen using a suitable colorimeter or spectrophotometer with the appropriate software (see AATCC Evaluation Procedure 7, Instrumental Assessment of the Change in Color of a Test Specimen).

8.2 Evaluation of staining.

8.2.1 Evaluate staining (see 12.10) as directed in Evaluation Procedure 2 using the Gray Scale for Staining or as directed in Evaluation Procedure 8 using the Chromatic Transference Scale. The scale used should be indicated when reporting the test results.

Grade 5—negligible or no color transfer.

Grade 4.5—color transfer equivalent to Step 4-5 on the Gray Scale for Staining or Row 4.5 on the 9-step AATCC Chromatic Transference Scale.

Grade 4—color transfer equivalent to Step 4 on the Gray Scale for Staining or Row 4 on the 9-step AATCC Chromatic Transference Scale.

Grade 3.5—color transfer equivalent to Step 3-4 on the Gray Scale for Staining or Row 3.5 on the 9-step AATCC Chromatic Transference Scale.

Grade 3—color transfer equivalent to Step 3 on the Gray Scale for Staining or Row 3 on the 9-step AATCC Chromatic Transference Scale.

Grade 2.5—color transfer equivalent to Step 2-3 on the Gray Scale for Staining or Row 2.5 on the 9-step AATCC Chromatic Transference Scale.

Grade 2—color transfer equivalent to Step 2 on the Gray Scale for Staining or Row 2 on the 9-step AATCC Chromatic Transference Scale.

Grade 1.5—color transfer equivalent to Step 1-2 on the Gray Scale for Staining or Row 1.5 on the 9-step AATCC Chromatic Transference Scale.

Grade 1—color transfer equivalent to Step 1 on the Gray Scale for Staining or Row 1 on the 9-step AATCC Chromatic Transference Scale.

8.2.2 The color transferred to the multifiber test fabric or bleached cotton test fabric square of 6.4.1 can be quantitatively determined by measuring the color difference between a piece of the original material and the stained material. Multifiber test fabrics No. 10A or FAA have bands of sufficient width to be within the aperture diameter capability of many colorimeters and spectrophotometers (see Evaluation Procedure 6, Instrumental Color Measurement and 12.14).

9. Interpretation of Results

9.1 Results from these tests are intended to approximate the color change effects (see 1.1) of five typical home or commercial launderings. These are accelerated tests, and in obtaining the required degree of acceleration some of the conditions, such as temperature, were purposely exaggerated. The tests have remained largely the same over many years while laundry detergents, washers and dryers, laundry practices and fabrics have changed (see monograph *"Standardization of Home Laundry Test Conditions,"* elsewhere in this TECHNICAL MANUAL). Consequently, caution in interpreting test results is advisable.

9.2 Test No. 1A—This test is for evaluating the colorfastness of textiles that are expected to withstand repeated hand laundering at low temperature. Specimens subjected to this test should show color change similar to that produced by five typical careful hand launderings at a temperature of 40 ± 3°C (105 ± 5°F).

9.3 Test No. 2A—This test is for ev
uating the colorfastness to washing
textiles that are expected to withstand
peated low temperature machine laund
ing in the home or in the commerc
laundry. Specimens subjected to this t
should show color change similar to t
produced by five commercial launderit
at 38 ± 3°C (100 ± 5°F) or by five ho
machine launderings at medium or wa
setting in the temperature range of 38
3°C (100 ± 5°F).

9.4 Test No. 3A—This test is for ev
uating colorfastness to washing of te
tiles considered washable under vigorc
conditions. Specimens subjected to tl
test should show color change similar
that produced by five commercial lau
derings at 49 ± 3°C (120 ± 5°F) or by fi
home machine launderings at 60 ± 3
(140 ± 5°F), both without chlorine.

9.5 Test No. 4A—This test is for eval
ating the colorfastness to washing of te
tiles laundered in the presence of ava
able chlorine. Specimens subjected
this test should show color change simi
to that produced by five commerc
launderings at 71 ± 3°C (160 ± 5°F) wi
1.9 L (2.0 qt) of 1% available chlori
per 45.4 kg (100.0 lb) of load (wh
wash formula) or by five home machi
launderings at 63 ± 3°C (145 ± 5°F) wi
3.74 g per L (0.50 oz/gal) of 5% availab
chlorine per 3.6 kg (8.0 lb) load.

9.6 Test No. 5A—This test is for eva
uating the colorfastness to washing
textiles that may be laundered in the pre
ence of available chlorine. Specime
subjected to this test should show col
change similar to that produced by fi
home machine launderings at 49 ± 3°
(120 ± 5°F) with 200 ± 1 ppm availab
chlorine.

10. Report

10.1 Report the test number.

10.2 Report the grade number dete
mined for color change in 8.1 and th
staining grade numbers for the evaluate
fibers in the multifiber test fabric and/
bleached cotton test fabric as determine
in 8.2.

10.3 State which scale (Gray Scale fo
Staining or AATCC Chromatic Transfe
ence Scale) was used in evaluating stain
ing (see 12.12).

10.4 Report the multifiber test fabri
used and if bleached cotton test fabri
was employed to avoid knit curling.

10.5 Report the detergent used wit
color change and staining results (se
12.6).

10.6 Report which laundering ma
chine is used.

11. Precision and Bias

11.1 Precision and bias statement

have been developed for Tests No. 2A and 5A. Although correlation work has been done, no precision and bias statements have been developed for Tests No. 1A, 3A and 4A.

11.1.1 Because of changes in the detergents used in this method, these precision and bias statements may not apply to data or information obtained with the currently available detergents.

11.2 Test No. 2A.

11.2.1 *Summary.* An interlaboratory test was carried out in May 1985 to establish the precision of Test No. 2A. A part of the test was to determine if the wider 15 mm (0.6 in.) No. 10A multifiber test fabric could be substituted for the 8 mm (0.33 in.) wide No. 10. The complete test consisted of six laboratories evaluating 10 materials in duplicate by one operator using Test No. 2A.

11.2.2 *Color Change.* Three raters from six laboratories independently evaluated nine materials in duplicate using the Gray Scale for Color Change. The components of variance as standard deviations of the colorfastness grades (averages of variances for No. 10 and No. 10A multifiber test fabrics) were calculated as follows:

Single-Operator Component	0.29
Within-Laboratory Component	0.29
Between-Laboratory Component	0.29

11.2.3 *Critical Differences.* For the components of variance reported in 11.2.2, two averages of observed values should be considered significantly different at the 95% probability level if the difference is equal to or exceeds the critical differences shown in Table II.

Table II—Critical Differences, Grades, for the Condition Noted[a]

No. of Observations	Single-Operator Precision	Within-Lab Precision	Between-Lab Precision
1	0.80	1.12	1.37
3	0.46	0.92	1.21
5	0.36	0.87	1.18

[a]The critical differences were calculated using t = 1.950 which is based on infinite degrees of freedom.

11.2.4 *Staining.* Three raters independently rated the six fibers of the multifiber test fabric (No. 10 and No. 10A) for 10 materials at six laboratories using the Gray Scale for Staining. Of the 60 possible fiber/fabric combinations, only 51 could be used. The components of variance were averaged for the No. 10 and No. 10A multifiber test fabrics and appear below as standard deviations of staining ratings:

Single Operator Component	0.27
Within-Laboratory Component	0.34
Between-Laboratory Component	0.25

11.2.5 *Critical Differences.* For the components of variance reported in 11.2.4, two averages of observed values should be considered significantly different at the 95% probability level if the difference equals or exceeds the critical differences shown in Table III.

Table III—Critical Differences, Grades, for the Condition Noted[a]

No. of Observations	Single-Operator Precision	Within-Lab Precision	Between-Lab Precision
1	0.75	1.20	1.39
3	0.43	1.03	1.25
5	0.33	1.00	1.22

[a]The critical differences were calculated using t = 1.950 which is based on infinite degrees of freedom.

11.2.6 *Bias.* Tests comparing five home launderings at 40°C (105°F) with one Launder-Ometer Test No. 2A indicate there is no bias between the two methods for the colorfastness and staining levels evaluated.

11.3 Test No. 5A, Chlorine Bleach.

11.3.1 *Summary.* An interlaboratory test was carried out in 1984 to establish the precision of Test No. 5A for determining the effect of chlorine bleach on the colorfastness of fabrics. All specimens were laundered in a Launder-Ometer by one operator. Color change in Test No. 5A was determined both visually and instrumentally. Details of the statistical analysis of the data can be found in the report, Third Interlaboratory Study of Proposed Launder-Ometer Test for Colorfastness of Fabrics to Chlorine and Non-Chlorine Bleaches, October 21, 1985, by J. W. Whitworth, Milliken Research Corp., Spartanburg, SC.

11.3.2 *Visual Assessment.* Four materials were tested at each of five laboratories. Three raters visually assessed the color change of four specimens. The components of variance as standard deviations of colorfastness grades were calculated as follows:

Single Operator Component	0.38
Within-Laboratory Component	0.28
Between-Laboratory Component	0.27

11.3.3 *Critical Differences.* For the components of variance in 11.3.2, two averages of observed values should be considered significantly different at the 95% probability level if the difference equals or exceeds the critical differences shown in Table IV.

Table IV—Critical Differences, Grades, for the Condition Noted[a]

No. of Observations	Single-Operator Precision	Within-Lab Precision	Between-Lab Precision
1	1.03	1.29	1.49
3	0.59	0.98	1.23
5	0.46	0.91	1.17

[a]The critical differences were calculated using t = 1.950 which is based on infinite degrees of freedom.

11.3.4 *Instrumental Assessment.* Color change as total color difference (CIELAB) was measured on a spectrophotometer or colorimeter using apertures ranging in size from 13-51 mm (0.5-2.0 in.) in diameter, illuminant D_{65}/10° observer or illuminant C/2° observer. Six materials were tested at each of six laboratories. One operator in each laboratory tested four specimens of each fabric. The components of variance for ΔE^* expressed as coefficients of variation were calculated to be:

Single-Operator Component	6.8%
Between-Laboratory Component	11.2%

11.3.5 *Critical Differences.* For the components of variance reported in 11.3.4, two averages of observed values should be considered significantly different at the 95% probability level if the difference equals or exceeds the critical differences shown in Table V.

Table V—Critical Differences, Percent of Grand Average for the Conditions Noted[a,b]

No. of Observations in Each Average	Single-Operator Precision	Between-Laboratory Precision
1	18.7	36.2
3	10.8	32.8
5	8.4	32.1

[a]The critical differences were calculated using t = 1.950 which is based on infinite degrees of freedom. [b]To convert the values of the critical differences to units of measure, multiply the critical differences by the average of the two specific sets of data being compared and then divide by 100.

11.3.6 *Bias.* Tests comparing five home launderings at 49°C (120°F) with one Launder-Ometer Test No. 5A indicate there is no bias between the two methods for the colorfastness levels evaluated (see 12.13).

12. Notes

12.1 The Launder-Ometer (a registered trademark of Atlas Electric Devices), is available with accessories from SDL Atlas L.L.C., 1813A Associate Lane, Charlotte NC 28217; tel: 704/329-0911; fax: 704/329-0914; e-mail: info@sdlatlas.com.

12.2 Teflon is a registered trademark of the DuPont Co., Wilmington DE 19898.

12.3 The preheater/storage unit may be a side unit to the laundering machine or a separate module with its individual electric heater and thermostat to control water bath temperatures for heating containers and solutions prior to loading the laundering machine.

12.4 Available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.

12.5 Multifiber test fabrics No. 1, No. 10 and No. 10A are available from Testfabrics Inc., P.O. Box 26, 415 Delaware St., W. Pittston PA 18643; tel: 570/603-0432; fax: 570/603-0433; e-mail: testfabric@aol.com. Multifiber test fabrics FA, FB and FAA are available from Textile Innovators Corp., div. of SDL Atlas L.L.C., P.O. Box 8, 101 Forest St., Windsor NC; tel: 252/794-9703; fax: 252/794-9704; e-mail: tic@sdlatlas.com. Bleached cotton test fabric in 32 × 32 ends × picks/cm (80 × 80 ends × picks/in.) construction, 100 ± 3 g/m² and without fluorescent whitening agent is available from both suppliers.

12.6 The 1993 AATCC Standard Reference Detergent WOB (without fluorescent whitening agent), a compact formulation, is the primary detergent to be used in this test method. Where the effect of a fluorescent whitening agent is to be evaluated, 1993 AATCC Standard Reference Detergent (with fluorescent whitening agent) should be used. Both detergents are available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.

12.7 Use distilled water or deionized water of not more than 15 ppm hardness to dissolve the detergent and for the test solutions.

12.8 Use sodium hypochlorite bleach purchased within the last six months for a stock solution.

12.8.1 To confirm the stock solution's hypochlorite activity, weigh 2.00 g liquid sodium hypochlorite into an Erlenmeyer flask and dilute with 50 mL of deionized water. Add 10 mL of 10% sulfuric acid and 10 mL of 10% potassium iodide. Titrate with 0.1*N* sodium thiosulfate until colorless.

Calculation:

% sodium hypochlorite

$$= \frac{(\text{mL } Na_2S_2O_3)(0.1N)(0.03722)}{(2.00 \text{ g NaOCl})} \times 100$$

The factor 0.03722 is derived by multiplying the molecular weight of NaOCl (74.45 g/mol) by 0.001 (mL to L conversion) and dividing by 2 (mols of thiosulfate per hypochlorite).

12.8.2 Oxidizing power of sodium hypochlorite is typically expressed in terms of available chlorine, the equivalent amount of diatomic chlorine present. A 5.25% NaOCl solution contains 50,000 ppm available chlorine.

12.9 Crockmeter test cloth, 32 × 33 ends × picks/cm (80 × 84 ends × picks/cm) combed cotton, desized, bleached (no fluorescent whitener or finishing material present) is available from the following suppliers: Testfabrics Inc P.O. Box 26, 415 Delaware St., W. Pittsto PA 18643; tel: 570/603-0432; fax: 570/60 0433; e-mail: testfabric@aol.com; Textile In novators Corp., div. of SDL Atlas L.L.C., P.C Box 8, 101 Forest St., Windsor NC; tel: 25 794-9703; fax: 252/794-9704; e-mail: tic sdlatlas.com.

12.10 If staining evaluations are needed f Tests No. 4A and 5A, they may be carried o using the corresponding Tests No. 2A or 3A which use no bleach. Test No. 2A is th no-bleach alternate for Test No. 5A, and Te No. 3A is the no-bleach alternate for Te No. 4A.

12.11 If multifiber test fabric is used in co junction with Tests 4A or 5A, the wool ca absorb the chlorine leaving very little f bleaching action. The wool may be remove from the multifiber test fabric before testing eliminate this effect.

12.12 For very critical evaluations and cases of arbitration, grades must be based the geometric Gray Scale for Staining.

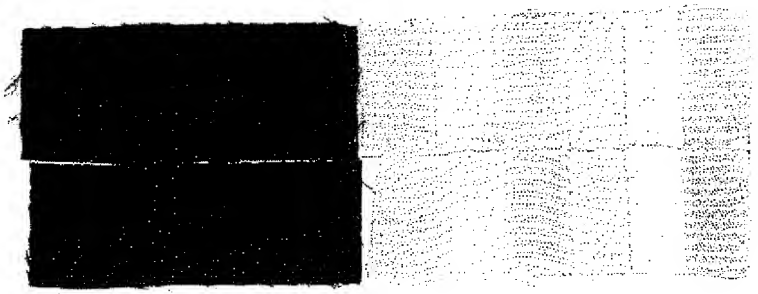
12.13 For additional information pertainir to the bias between Test No. 5A and five hon washes, refer to Fig. 1 in Interlaboratory Stu of Proposed Launder-Ometer Test for Colo fastness of Fabrics to Chlorine and Non-Chl rine Bleaches, Report to AATCC Committ RA60, Colorfastness to Washing Test Metho November 1984, New York NY by L. Farmer and J. W. Whitworth of Millik Research Corp., Spartanburg SC, and J. G. Te AATCC Technical Center, Research Triang Park NC.

12.14 AATCC Evaluation Procedure gives a method for computing Gray Sca grades from color measurement data.

Colorfastness to Laundering, Home and Commercial: Accelerated

AATCC Test Method 61-2003 Test Number 2A

Dyestuff 5 according to the present invention



Dyestuff 6 according to Example 20 of GB 2,104,088 to Dawson et al. (prior art)

DyStar Frankfurt 20/11/2007